

Nano Fabrication of Oxide Patterns Using Atomic Force Microscopy on Titanium: Towards the Development of Nano Devices

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Abstract—In this paper related to the field of nano technologies, we report on nano lithography for the fabrication of oxide patterns on titanium thin film using Atomic Force Microscopy (AFM). The patterns consist of lines, dots, and surfaces. Their morphology is assessed in terms of width and height as a function of the growth conditions, e.g., for lines, bias voltage and sweep velocity, and for dots bias voltage and duration of application of the bias voltage. We show that the results present a satisfactorily reproducibility. This opens the way to further developments: nano devices among which simple and double junctions are currently in development, the final goal being the development of single electron devices (SETs).

Keywords—Atomic force microscope; Nano lithography; Ti/TiOx, Nano devices

I. INTRODUCTION

For more than 4 decades, Complementary Metal Oxide Semiconductor (CMOS) technology has enabled a tenfold increase in computing performance every 5 years. Throughout that period, this trend has mainly been achieved by scaling down MOS devices. Nowadays it is threatened by ever increasing power dissipation of integrated circuits. Among competing emerging research devices, we can find “the one electron electronics” in which the elementary block is the Single Electron Transistor (SET), which features particularly low power consumption [1]. The SET is a three terminal switching device which conveys electrons through successive tunnel events between two electrodes separated with one conductor island (Figure 1). Because its way of working lies in the controlled transport of electrons one by one, the SET is the great winner in terms of electrical consumption comparatively to the others emergent devices.

The realization of an island in such devices brings the necessity of creating tunnel junctions. For electron tunneling to occur in a controlled manner, it is crucial to control distances at a nanometer scale. This has been carried out using various techniques such as break junctions [2], electromigration [3], electrodeposition [4], scanning tunneling microscopy [5], electron beam lithography/shadow evaporation (alone or associated with other techniques like Chemical-Mechanical Planarization) [6][7], nanoparticle positioning [8] and nanoscale oxidation [9][10][11][12]. However, to our knowledge, no systematic study enabling

systematic parameter study and derivation have been completed using the latter process when applied to the fabrication of components. Moreover, this technology, which enables “drawing” the mono-electronic component on a thin metal layer, presents the advantage of high flexibility in terms of dimension variability.

This AFM direct writing technique enables the processing of electronic components of increasing complexity (nanowires, nanojunctions, monoelectronic components like SETs), with a possibility of varying the geometry. However this flexible technology involves many parameters (nature of the AFM tip as well as voltage applied, duration, humidity, temperature) which need to be assessed. This is the goal of the present work.

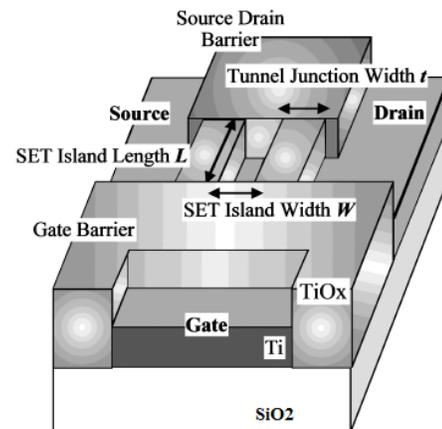


Figure 1. Illustration of the topography of a SET [10].

This paper will be divided in three parts, the first one will be related to the protocol and the equipment used at the laboratory to obtain our results, the second one will show our work on oxide characterization using three main parameters like height of the oxide, width of the oxide and the reproducibility of the method. In the last part, we will discuss of our results and the future perspectives of our work.

II. PROTOCOL AND EQUIPMENTS

All measurements reported in this paper were made using an Atomic Force Microscope NT-MDT Integra. The tips used were conductive tips Nanosensor PPP-EFM Silicon coated with 25 nm of PtIr. The force constant was about 2.8 N/m and the resonance frequency is comprised between 45 and 115 kHz. The resonance frequency is very important in this case because all the experiments were made using the semi-contact mode of the AFM.

In order to regulate the humidity in the microscope chamber, we used two channels of nitrogen. One goes through a water bubbler in order to add some humidity to the nitrogen introduced in the microscope chamber, the other not. Thanks to this set-up the humidity rate was constantly maintained at 55%.

The samples studied were originally silicon substrates with a thermal oxide layer of thickness 520 nm. Then we deposited 5 nm of Ti using e-beam assisted evaporation. As the samples were stored in an ambient environment, a native oxide layer of about 2 nm arose on the Ti layer (as measured by ellipsometry). The roughness parameter Rq or RMS after the Ti deposition reached 0.2 nm.

III. OXIDE CHARACTERIZATION

The working principle of the method implemented here to fabricate nano devices relies on the oxidation using AFM, works as follows.

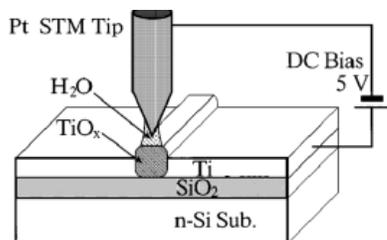


Figure 2. Schematic of the oxidation process with an AFM tip [9].

In ambient environment we can find a thin layer of water on every surface due to humidity contained in the air. When the tip of the AFM approaches the surface, a meniscus of water is formed around the tip. Then, if a bias voltage is applied to the tip, a current carried by the oxyanions comes up, which leads to an oxido-reduction reaction between the oxyanions and the metallic surfaces and proceed to the growth of TiO_x as shown on the Figure 2.

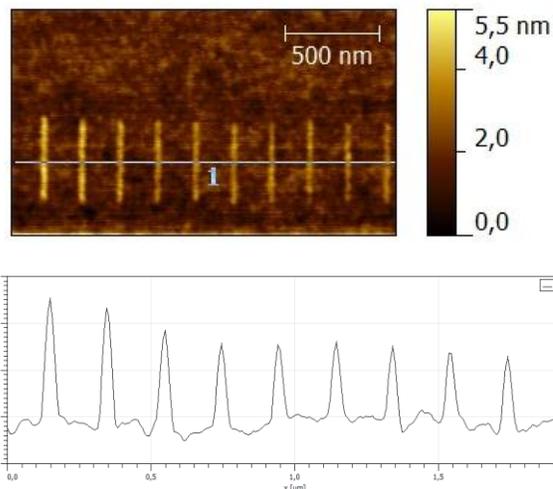


Figure 3. AFM image and corresponding line scan showing oxides lines fabricated using different sweep velocities ($0.4\mu\text{m/s}$ -> $2.0\mu\text{m/s}$ per step of $0.4\mu\text{m/s}$ every two lines) with a bias voltage of -6V .

First, we evaluated the geometrical properties of oxide lines by measuring their height and protrusion width (FWHM), as this method takes into account the topology of the AFM tip. These parameters have been extracted using the average of the profile of the oxide lines as shown on the Figure 3. The evolution of these parameters is assessed as function of the bias voltage applied on the tip and the sweep speed of the tip, or the time per dots in the case of the oxidation of table of dots.

A. Characterization of the oxide height

The experimental height of the oxide lines measured as a function of the bias voltage of the AFM tip is reported in Figure 4. This graph displays also the dependency of the oxide height on the sweep velocity of the AFM probe. The tendencies observed here are in agreement with those of the literature [11]: the oxide height decreases sharply with the absolute value of the bias voltage and also, but to a lesser extent, for increasing sweep velocity.

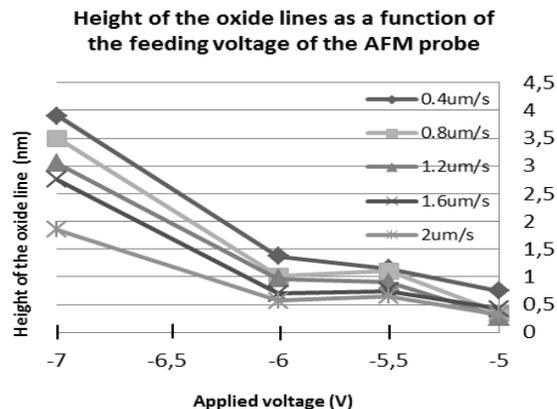


Figure 4. Experimental height of the oxide lines as a function of the bias voltage (comprised between -5V and -7V).

B. Characterization of the oxide width

Figure 5 shows how the full width at half maximum (FWHM) of the lines varies depending on the bias voltage and on the sweep velocity.

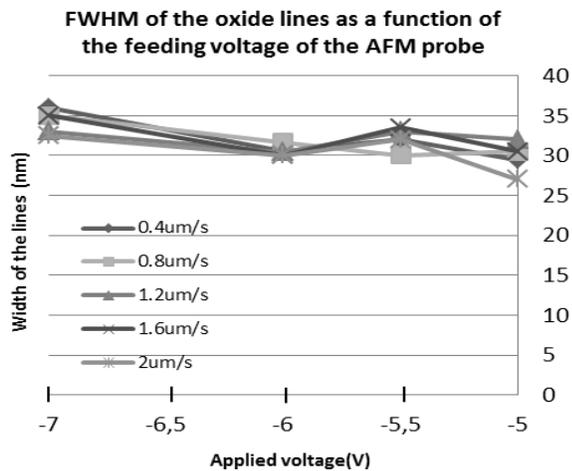


Figure 5. Experimental FWHM of the oxide lines as a function of the bias voltage (comprised between 5V and -7V).

The line width is found to remain roughly constant over the whole span of parameters tested here. The small discrepancies measured belong indeed to the experimental uncertainty which is estimated to ± 8 nm, the image resolution being $256 \text{ pixels} \times 256 \text{ pixels}$ for a scan length reaching $2 \mu\text{m}$.

C. Process reproducibility

The process reproducibility has been assessed by generating arrays of 36 oxide dots. For the results shown in Figure 6, the voltage applied was respectively -6V and -7V. In both cases the duration of the applied voltage reached 200 ms/point. For comparison, the oxide lines considered in the preceding paragraph were performed with scanning velocities ranging between $2 \mu\text{m/s}$ and $0.4 \mu\text{m/s}$, which correspond to a duration of applied voltage ranging from 4 ms/point to 20 ms/point.

Table 1 summarizes the measured height and FWHM of the dots. The standard deviation for either the dot height or FWHM remains very low. This attests for a good reproducibility of the oxidation process.

TABLE I. EXPERIMENTAL HEIGHT AND FWHM OF OXIDE DOTS

Voltage applied	FWHM (nm)		Height (nm)	
	Average	Standard deviation	Average	Standard deviation
-7V	42.5	3.7	4.5	0.5
-6V	36	3.2	2.4	0.5

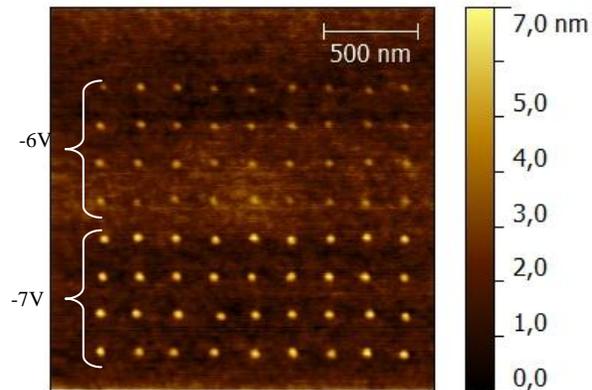


Figure 6. Arrays of oxide dots performed at a bias voltage of -7V and -6V respectively. The duration of the applied voltage is 200 ms/point.

Note that the averaged values reported for the height are slightly exceeding those reported in the figures 4 and 5 and confirm the tendency of increasing the oxide height with the oxidation time.

IV. CONCLUSION AND FUTURE WORK

In this work dedicated to nano lithography, we evaluated the influence of the applied voltage and of the sweep velocity when fabricating oxide patterns using AFM. The oxide patterns consist in lines, dots, and surfaces. Their width and height, which depend on the growth conditions, were assessed as well as the process reproducibility. The height of the lines increases with the absolute value of the bias voltage and with decreasing sweep velocity for lines (or increasing duration of the voltage applied per point for dots). On the other hand, the line width is not parameter-sensitive in the range evaluated.

The immediate outlook of this study is the processing of nano devices of increasing complexity, among which nanowires, simple junctions, double junctions, the final goal being the development of single electron devices (SETs). As a first example of nano device, Figure 7 illustrates a nanowire-type structure. A Ti-nanowire, having a width of 20 nm arises between 2 surfaces of μm -size of locally anodized Ti.

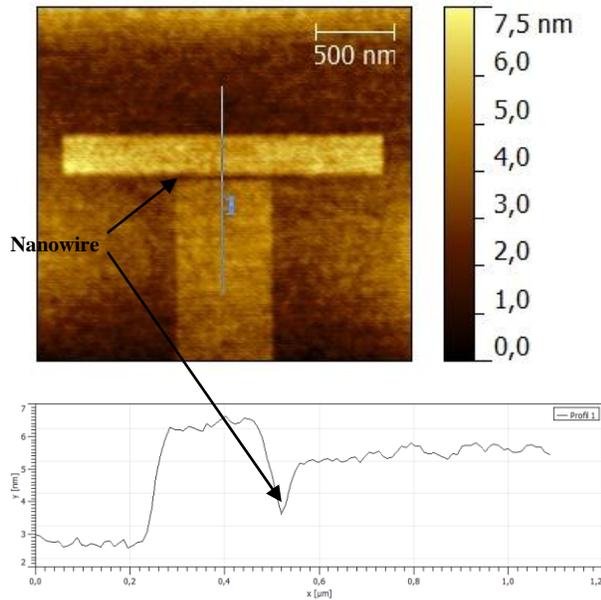


Figure 7. Example of a nano-wire processed by local anodization.

This first demonstration opens the way to more sophisticated devices which will be further developed in the oral talk.

V. ACKNOWLEDGMENTS

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